Perfood Newsletter

Issue 3: September 2012

Dear reader,

I am very pleased to introduce to you the third PERFOOD newsletter. The PERFOOD project is nearing its formal end (30 November 2012). The preparations are ongoing for a dissemination workshop which will also take place in November 2012 in Idstein, Germany during the 4th International Workshop on PFAS. An announcement can be found at the last page of this newsletter and on the Perfood website.
In this issue you will find the results of the 5th Interlaboratory study on perfluoroalkyl substances (PFAS) in food and environmental samples, conducted in the framework of work package 2 of the PERFOOD project.
The results of the surveys performed in the course of the PERFOOD project are discussed in a contribution from the leaders of work packages 3 and 6. The results show that the intake of PFAS across Europe through our diet is in general well below existing standards (in so far as these are available) and that distinct the geographical differences exist between contributions from food items to PFAS intake in the selected North, South, East and West European Regions .
The results of the PERFOOD project will be submitted to the EFSA and NORMAN data bases. PERFOOD is now formally linked to research projects in Belgium, Norway and China that investigate the sources and exposure to PFAS in the general population in these countries.

On behalf of the PERFOOD consortium,

Pim de Voogt
Coordinator PERFOOD

THE 5th INTERLABORATORY STUDY ON PERFLUOROALKYL SUBSTANCES (PFAS) IN FOOD AND ENVIRONMENTAL SAMPLES

by
Jana Weiss & Ike van der Veen

Perfluoroalkyl substances (PFAS) are omnipresent in food and in the environment. To study the distribution of these chemicals in the environment and to assess the human and environmental exposure, many laboratories have developed methods for analysis of PFAS in food and environmental matrices. To assess the intercomparability of the data produced by those laboratories, and to follow-up on earlier interlaboratory studies (ILSs) to evaluate possible improvements, the 5th ILS on PFAS in food and environmental samples was organized in 2011 as part of the PERFOOD project. Since food and beverage matrices are especially challenging owing to low levels, the study focused on the analytical challenge of determining PFAS concentrations at the low pg/g and ng/L concentrations.

In total 29 of the 31 registered laboratories submitted data in the study on the analysis of PFAS in food, beverage and environmental samples (pig liver, drinking water, fish, and vegetables), but since participating laboratories were able to choose between packages of samples the number of participating laboratories varied per sample. The participants used their in-house methods for analysis of the test materials. Standard solutions with PFAS in undisclosed concentrations were also analysed by the participants to check their calibration procedures. The results were collected and statistically evaluated using the 'Cofino statistics'. Between lab coefficients of variation (CVs) and z-scores were appointed to the laboratory's results as an expression of accuracy.
The full report has been distributed to the participants and the summary published elsewhere [1]. The between laboratory CVs are below 50% for the majority of the matrices and compounds analysed (Figure 1). The higher CVs can be attributed compounds at low levels or matrices with challenges to analyse (e.g. pig liver).

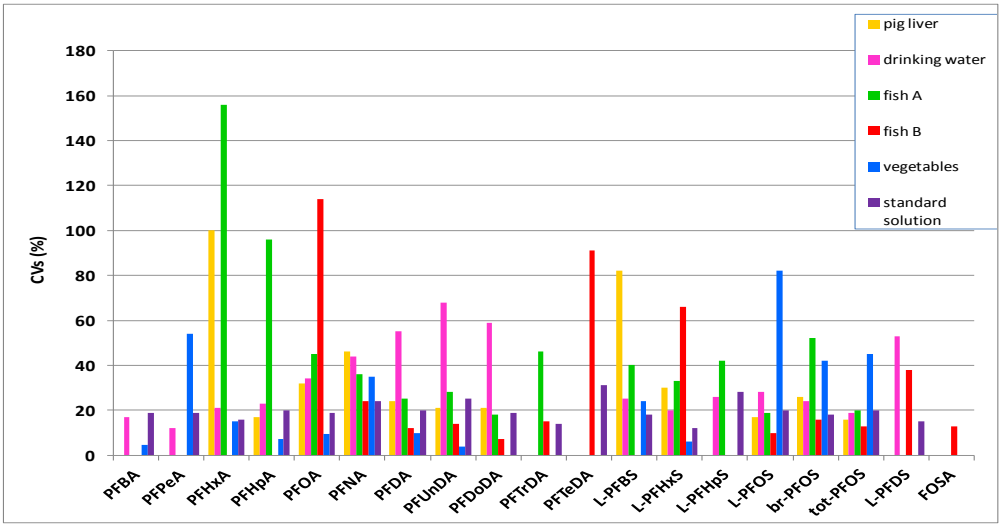

Figure 1. Group performance expressed as coefficient of variation (CV) between all laboratories for different PFAS in all matrices.

Twenty-one participant analyzed the pig liver. Seven PFAS congeners could be given an assigned value. PFBA (0.34 ng/g ww) and PFPeA (0.29 ng/g ww) concentrations were the highest reported, but the precisions were low with CVs of 135% and 246%, respectively. The L-PFOS determination was more successful, the assigned value was 0.24 ng/g ww, with 50% of the laboratory receiving a satisfactory z-score and the between laboratory CV of 17%. The remaining PFAS concentrations reported were below 0.1 ng/g ww. Satisfactory z-scores (i.e. $Z \leq |2|$) was reached for three compounds, PFNA (29%), linear (L)-PFOS (50%), and branched (br)-PFOS (57%).
Two fish muscle tissue materials, A and B, were distributed. Fish A contained lower PFAS concentrations than fish B. The between laboratory CVs obtained for sample B were equal or better than for sample A (Figure 1), and a higher fraction of the

participating laboratories (31-100% depending on the determined compound) obtained satisfactory z-scores for the B sample, compared to A (23-75% depending on the determinand). The analytical performance for the fish muscle tissue was better in the current study compared to a study from 2009 [2], in which the same fish sample (B) was used (Figure 2).

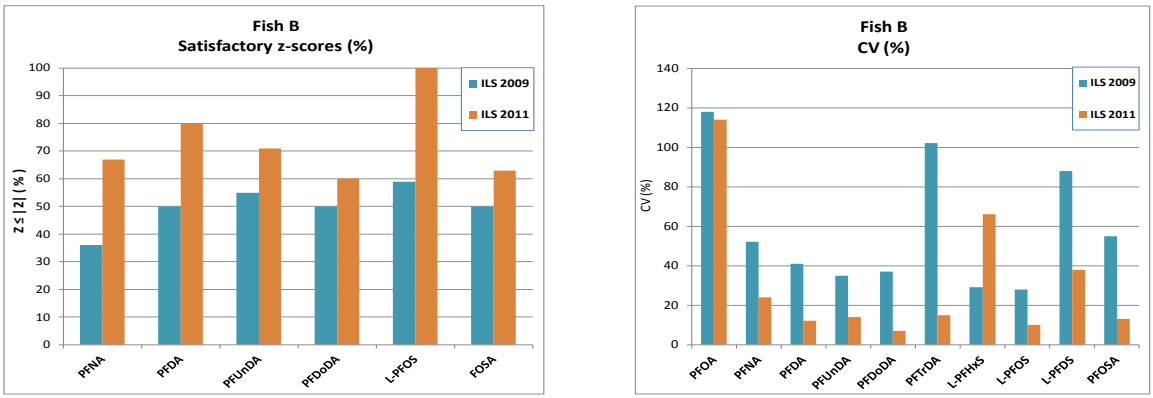

Figure 2. Comparison of group performance of ILS 2009 and ILS 2011 for different PFAS in fish muscle tissue B expressed as % satisfactory z-scores (i.e. Z≤|2| (left) and coefficient of variation (CVs, right).

The PFAS concentrations in the vegetable mixture item analyzed in this ILS were all below the ng/g ww level (except PFBA). The between laboratory CVs for the most abundant congeners, perfluorocarboxylic acids (PFCA), were satisfactory (<25%), except for PFPeA (54%) and PFNA (35%). The CVs for PFOS (L-, br and tot-) were between 42 and 82 % and this reflects the challenge to analyze concentrations below 0.050 ng/g ww. The percentage of satisfactory z-scores was high for the vegetable sample (80-100%), probably due to the limited number of participants. Only the PERFOOD members (n=5) analyzed the vegetable matrix. Satisfactory z-scores were obtained from 25-71% (depending on the determinand) of the laboratories that submitted results for the drinking water sample. Comparison of the water sample used in the study of 2009 (freshwater taken from a canal close to Amsterdam) with the water sample of the current study (tap water) is shown in Figure 3. The percentages of satisfactory z-scores were similar between the sets, despite the fact that the tap water from 2011 contained lower PFAS concentrations compared to the 2009 water sample. It can be expected that the analytical challenge is lower with a cleaner matrix such as tap water, but as well that the accuracy is easier to perform with elevated concentrations. The between laboratory CVs improved significantly between the two ILS occasions.

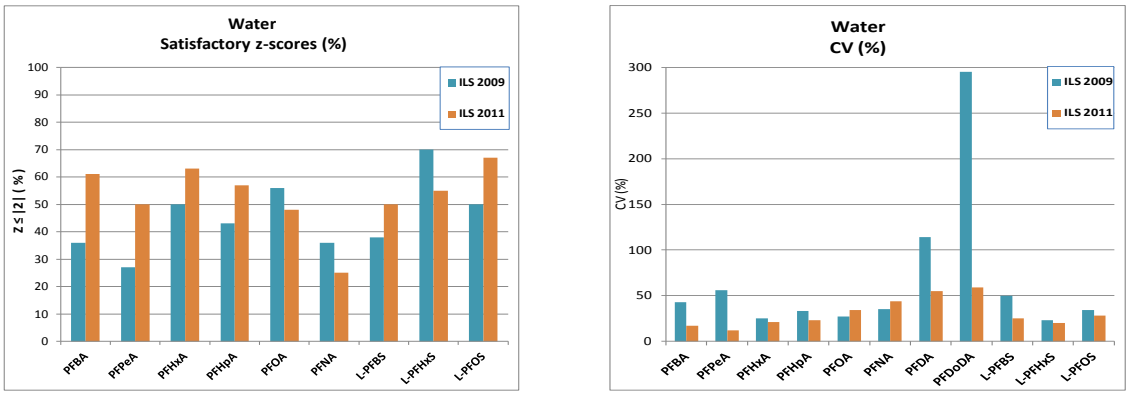

Figure 3. Comparison of group performance of ILS 2009 [5] and ILS 2011for different PFAS in water expressed as % satisfactory z-scores (i.e. Z≤|2| (left) and coefficient of variation (CVs, right).

The performance for the standard solution was less satisfactory than in 2009. In the current study 34-62% of the laboratories, depending on the determinands, obtained satisfactory z-scores, with an average of 47%. In the interlaboratory study of 2009 40-86% with an average of 74% obtained satisfactory z-scores (Figure 4). Again, the 7-16 fold higher PFAS a concentration in the standard solution in the study of 2009 is a reasonable explanation for the decrease in laboratories obtaining satisfactory z-scores in 2011. This could indicate that analytical challenges are still an issue regarding low concentrations (ng/mL), although all determinands (except PFTeDA) between laboratory CVs are below 25%, which is considered satisfactory (Figure 4).

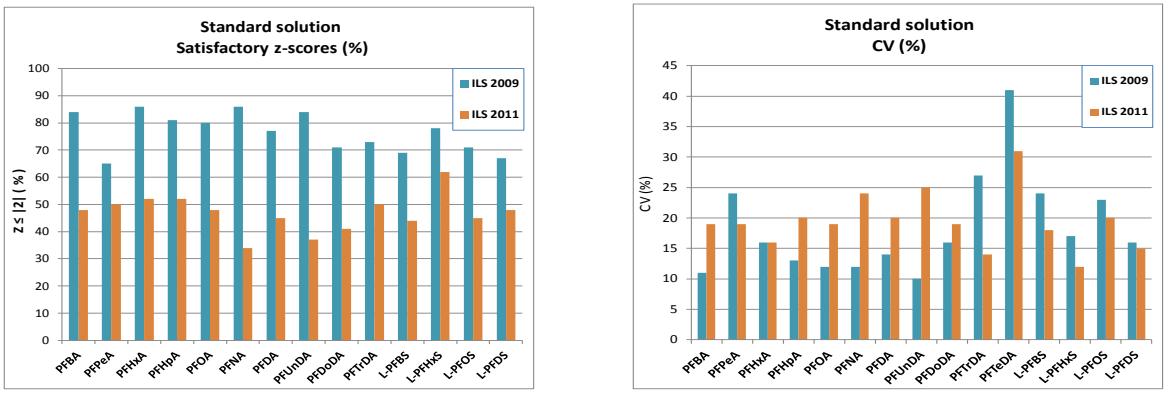

Figure 4. Comparison of group performance of ILS 2009 [5] and ILS 2011 for different PFAS in standard solution expressed as % satisfactory z-scores (i.e. Z≤|2| (left) and coefficient of variation (CVs, right).

Comparison of the average of absolute z-scores per matrix obtained by PERFOOD partners with the average of absolute z-scores obtained by other participants showed that PERFOOD partners performed within the same range as other participants. Despite the improved analytical performance, there are sources of variance still to be considered. The participants were encouraged to report

the linear isomer values instead of the sum for PFBS, PFHxS, PFHpS and PFOS, and in addition the branched and sum of the PFOS isomers. More than one third of the participants (37%) still reported the sum of the isomers and this contributed to the variation in the reported values. In addition, the reported PFOS concentrations in fish may be overestimated by some participants due to the presence of taurodeoxycholic acid (TDCA). TDCA needs special attention to be correctly chromatographically separated from PFOS, or by MS, which was not taken care of by 24% of the participants.
In conclusion – considering the low concentrations present in the test materials -improvements can be reported regarding the analytical performance of PFAS in food and environmental samples.

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2. Van Leeuwen, S.P.J., Strub, M., Cofino, W., Lindström, G., and van Bavel, B. Institute for Environmental Studies (IVM), VU University, Amsterdam. Report nr.-11/04 (2011)

PERFOOD: THE RESULTS ACHIEVED

by
Gianfranco Brambilla & Dorte Herzke

The scientific activities of the EU PERFOOD Project are coming to the end and now, and the extend and the geographical differences of Perfluoros intake across the selected North, South, East and West European Regions are becoming more clear (Norway, Italy, Belgium, and Czech Republic). A considerable number of different PFAS were investigated and PERFOOD was able to detect and evaluate additional compounds besides PFOS and PFOA due to the application of dedicated especially developed analytical methods.

The estimated intakes resulted of no concern for the general population, adults and children: the mean food intake for PFOS and PFOA result largely below (2-3 orders of magnitude) the TDI of 150 and 1,500 ng kg bw, respectively, stated in the EFSA 2008 Opinion (Figure 1) for i) single raw food items, ii) ready-to-cook uncooked food items industrially produced, iii) cooked whole meals reflecting different food habits and age groups. Single raw food items from hot-spots were investigated as well for worst-case-scenario-evaluations.

Anyway regional differences were noted with respect both on a different contamination pattern found in the food items sampled and on the different food consumption habits (Figure 2).

It is worth noting that the EU PERFOOD sampling design allowed to discover the occurrence of PFOA in some vegetables, not considered in previous intake studies, besides the overall comparability of similar food items collected in four different regions of Europe).

The consistency of the overall EU PERFOOD intake estimate is supported by the analytical results from composite foods, meals (sampled at canteen and fast food level) and duplicate diets. In all these “ready to

eat" servings the found contamination is in good agreement with that computed on the deterministic basis. The minor differences noted for PFOS and PFOA may be attributed to wild fish servings and the use of bottled instead of tap water (Table 1). However, the consumption of some food items from hotspot areas may result in intake estimates that exceeds the established TDI. This stresses the necessity to use in such areas a more integrated approach, to deepen the link between the environmental quality and the health determinants.

The EU PERFOOD food intake estimates suggest that the internal doses reported in the bio-monitoring studies may account also from non food exposures in the recent past, as those deriving by the large use of perfluoros in EU consumers products, till 2000.

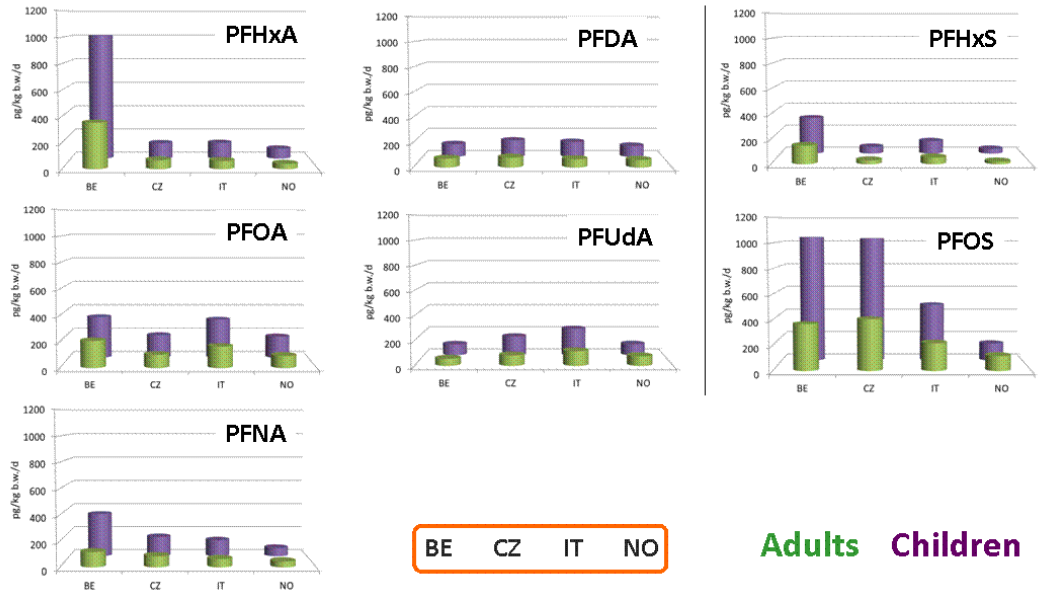

Figure 1: overview of estimated averaged intakes (medium bound), expressed as pg/kg body weight per day, in the selected European regions.

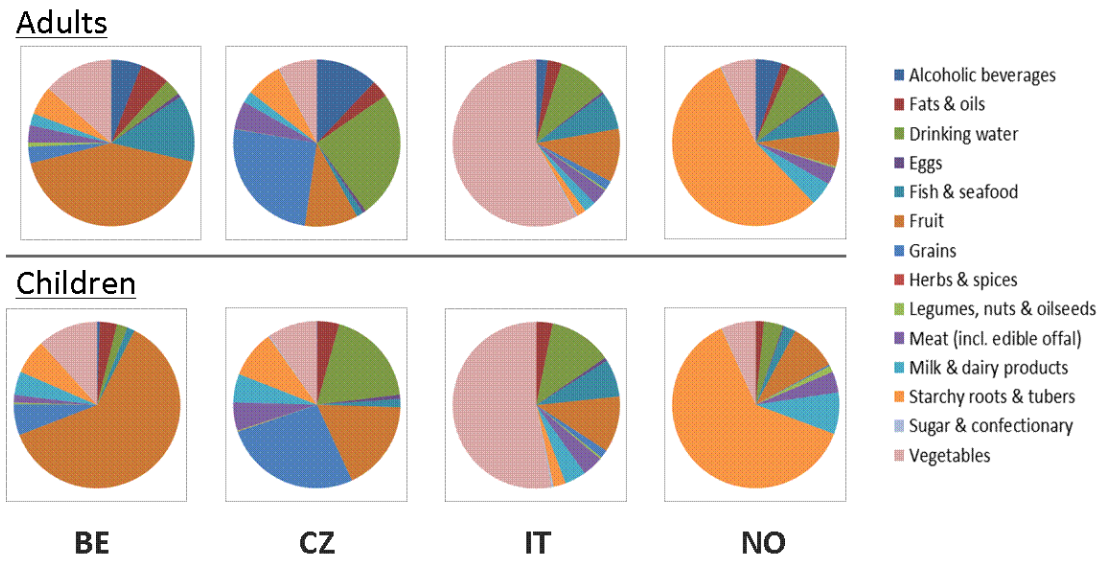

Figure 2: relative contribution of the different food groups to PFOA intake, in the selected European Regions.

Towns	PFHxA			PFOA			PFNA			PFDA		
	A	LB	UB	A	LB	UB	A	LB	UB	A	LB	UB
Brescia	*12.5*	2.5	8.6	*6.00*	11.2	15.9	*6.00*	2.5	9.0	8.20	0	7.8
Ferrara	*12.5*	4.1	9.6	6.50	13.2	18.7	*6.00*	0.65	7.4	*6.00*	0	7.7
Perugia*	*12.5*	2.9	9.6	*6.00*	10.5	14.6	*6.00*	3.1	9.8	*6.00*	0.2	8.0
Portici (Neaples)*	*12.5*	3.4	9.4	7.40	9.75	13.6	6.30	2.0	8.2	*6.00*	0.1	7.5
Genoa 1*	*12.5*	3.6	8.4	*6.00*	7.16	11.9	*6.00*	3.1	8.5	8.10	0.003	6.4
Genoa 2*	*12.5*	2.2	4.3	*6.00*	6.93	12.5	*6.00*	3.0	5.7	*6.00*	0.1	3.7

Towns	PFUnDA			PFHxS			PFOS L			PFOS B		
	A	LB	UB	A	LB	UB	A	LB	UB	A	LB	UB
Brescia	*6.0*	0	15.6	*6.0*	1.1	7.7	13.9	3.4	5.8	*6.0*	0.11	4.4
Ferrara	*6.0*	0	16.7	*6.0*	0.1	7.8	16.3	1.0	4.4	*6.0*	0.06	3.9
Perugia*	*6.0*	0	12.8	*6.0*	1.4	6.4	*6.0*	2.8	4.5	*6.0*	0.17	3.2
Portici (Neaples)*	*6.0*	0.06	13.0	*6.0*	1.7	6.7	24.7	2.7	4.6	7.1	0.20	3.2
Genoa 1*	*6.0*	0.003	12.1	*6.0*	1.5	6.3	15.9	3.3	5.0	*6.0*	0.24	2.8
Genoa 2*	*6.0*	0.04	0.38	*6.0*	0.6	4.3	*6.0*	1.9	3.5	*6.0*	0.19	2.3

A = analytical result | LB = lower bound estimate | UB = upper bound estimate | *italics = Limit of determination* | **bold = determined values**

Table 1: PFAS contamination (pg/g) (A) recovered in the weekly school canteen meals served in five Italian towns. Analytical values compared with the estimates recovered from meal composition and PFAS occurrence in the correspondent single food items

PERFOOD PUBLICATIONS AND CONFERENCE PROCEEDINGS

Contents lists available at SciVerse ScienceDirect

Journal of Chromatography A

journal homepage: www.elsevier.com/locate/chroma

A matrix effect-free method for reliable quantification of perfluoroalkyl carboxylic acids and perfluoroalkane sulfonic acids at low parts per trillion levels in dietary samples

Robin Vestergren[1], Shahid Ullah[2], Ian T. Cousins[3], Urs Berger*

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Reviews of Environmental Contamination and Toxicology 208, 179-215. DOI 10.1007/978-1-4419-6880-7_4.

Perfluorinated Substances in Human Food and Other Sources of Human Exposure

Wendy D'Hollander, Pim de Voogt, Wim De Coen, and Lieven Bervoets

Environmental Science & Technology

Article

pubs.acs.org/est

Impact of Treatment Processes on the Removal of Perfluoroalkyl Acids from the Drinking Water Production Chain

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Proceedings 32nd International Symposium on Halogenated Persistent Organic Pollutants 26-31 Aug 2012 Cairns (AUS)

INTAKE OF SELECTED PFAS IN THE ITALIAN GENERAL POPULATION

Dellatte E[1], Brambilla G[1]*, De Filippis SP[1], di Domenico A[1], Hertze D[2], Hajslova J[3], Eschauzier C[4], D'Hollander W[5], Heinemeyer G[6], Klenow S[6], and De Voogt WP[4]

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Journal of Chromatography A, 1218 (2011) 4312–4321

Contents lists available at ScienceDirect

Journal of Chromatography A

journal homepage: www.elsevier.com/locate/chroma

Simple, high throughput ultra-high performance liquid chromatography/tandem mass spectrometry trace analysis of perfluorinated alkylated substances in food of animal origin: Milk and fish

Ondrej Lacina, Petra Hradkova, Jana Pulkrabova, Jana Hajslova*

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Talanta 86 (2011) 329–336

Contents lists available at SciVerse ScienceDirect

Talanta

journal homepage: www.elsevier.com/locate/talanta

Analysis of perfluorinated phosponic acids and perfluorooctane sulfonic acid in water, sludge and sediment by LC–MS/MS

X. Esparza[a], E. Moyano[a], J. de Boer[b], M.T. Galceran[a], S.P.J. van Leeuwen[b,*,1]

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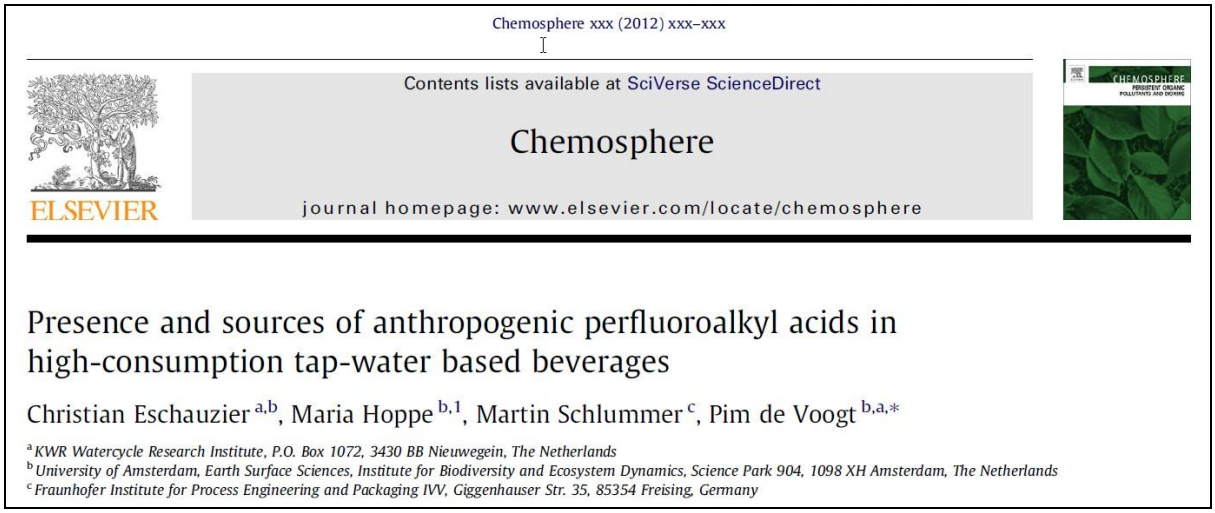

Chemosphere xxx (2012) xxx–xxx

Contents lists available at SciVerse ScienceDirect

Chemosphere

journal homepage: www.elsevier.com/locate/chemosphere

Presence and sources of anthropogenic perfluoroalkyl acids in high-consumption tap-water based beverages

Christian Eschauzier [a,b], Maria Hoppe [b,1], Martin Schlummer [c], Pim de Voogt [b,a,*]

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Robin Vestergren, Shahid Ullah, Ian T. Cousins, Urs Berger, 2012. A matrix effect-free method for reliable quantification of perfluoroalkyl carboxylic acids and perfluoroalkane sulfonic acids at low parts per trillion levels in dietary samples. J Chromatogr. A, 1237: 64-71, 2012

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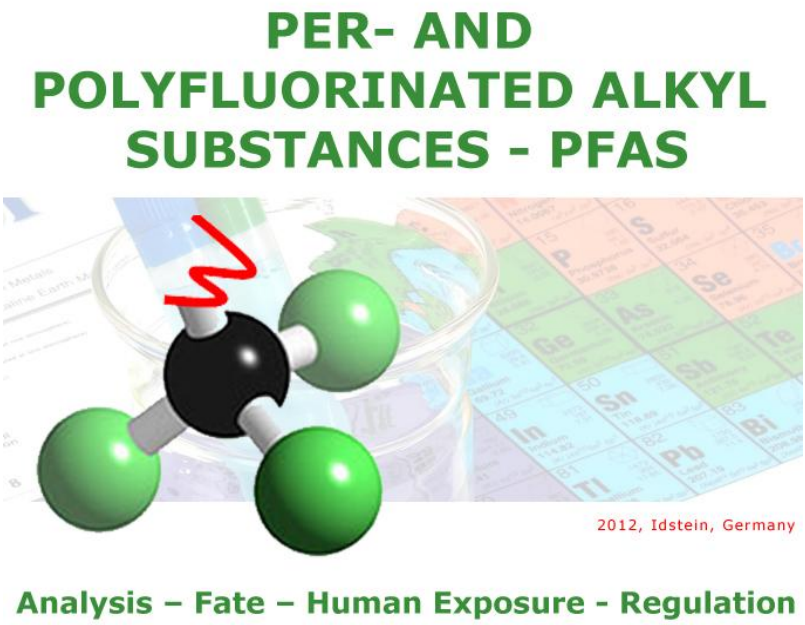
Announcement

4th INTERNATIONAL WORKSHOP on
Per- and Polyfluorinated Alkyl Substances – PFAS

November 7-9, 2012, Idstein, Germany



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